# Purple wilfordii insecticide and its preparing process

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### Abstract of CN1317248

An insecticide "Zileiteng" contains TW1, TW2 and TW3 in weight ratio of 1:(0.8-1.2):(0.8-1.2), which are extracted from the raw wilfordii through seeping, silica gel column chromatography and eluting. Its advantages include simple preparing process, high stability and no environmental pollution.

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A MILLETTIA ATROPURPUREA<sup>1</sup> PESTICIDE AND PREPARATION METHOD THEREOF

[54] Title of Invention: A Millettia Atropurpurea Pesticide and Preparation Method Thereof

[57] Abstract. This invention discloses a green pesticide whose main component includes extracts TW<sub>I</sub>, TW<sub>II</sub> and TW<sub>III</sub> of the raw medicinal herb Tripterygium wilfordii Hook f. at a weight ratio of TW<sub>I</sub>: TW<sub>II</sub>: TW<sub>III</sub> equal to 1:0.8~1.2:0.8~1.2. Alternatively, the principal components include TW<sub>I</sub>, TW<sub>II</sub> and TW<sub>III</sub>. The described preparation method involves taking the raw herb Tripterygium wilfordii Hook f., percolating, extracting it and eluting on a silica gel column for analysis and separation, thus obtaining the extract TW<sub>I</sub>. Alternatively, the raw herb Tripterygium wilfordii Hook f. is taken, percolated, extracted and eluted with different liquid cluants, thus collecting the components TW<sub>II</sub> and TW<sub>III</sub>. Therefore this is a pesticide with a straightforward technological process; it is a stable product and a pesticide harmless to the public.

#### **CLAIMS**

- 1. A green pesticide whose main component includes extracts  $TW_I$ ,  $TW_{II}$  and  $TW_{III}$  of the raw medicinal herb Tripterygium wilfordii Hook f. at a weight ratio of  $TW_I$ :  $TW_{III}$  equal to 1:0.8~1.2:0.8~1.2.
- 2. A green pesticide whose main component includes extracts TW<sub>II</sub>, TW<sub>II</sub> or TW<sub>III</sub> of the raw medicinal herb Tripterygium wilfordii Hook f.
- 3. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that raw medicinal herb Tripterygium wilfordii Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with chloroform to obtain a chloroform extract and subjected to silica gel column chromatography, then eluted with mineral ether: ethyl acetate to obtain the extract TW<sub>I</sub>.
- 4. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that raw medicinal herb Tripterygium wilfordii Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with ethyl ether to obtain an ethyl ether extract, subjected to silica gel column chromatography, then eluted with ethyl ether, to collect fractions containing TW<sub>II</sub> and TW<sub>III</sub>, which then are separately combined, again subjected to silica gel column chromatography, and eluted with chloroform: acetone to collect components TW<sub>II</sub> and TW<sub>III</sub>.

<sup>&</sup>lt;sup>1</sup> This translation is an unsupported guess. The combination 紫雷藤 (zileiteng) gets only 3 hits on Google and the plant is referred in the text of the patent under a different name 雷公藤 (leigongteng) for which the translation is Tripterygium wilfordii Hook f. – Translator's note.

5. The method of preparing the green pesticide described in Claim 1 or Claim 2 characterized in that when the extract of the raw medicinal herb Tripterygium wilfordii Hook f. is taken to prepare the pesticide, the content ratio of the TW<sub>I</sub>, TW<sub>II</sub> and TW<sub>III</sub> extracts is 1:0.8~1.2:0.8~1.2, and the content of the three components TW<sub>I</sub>, TW<sub>II</sub> or TW<sub>III</sub> in the extract is 1/500.

## Specifications

A Millettia Atropurpurea Pesticide and Preparation Method Thereof

This invention pertains to a plant pesticide and preparation method thereof.

Tripterygium wilfordii Hook f. (Celastracae) is a Tripterygium woody climber plant. Its main medicinal parts are roots, leaves, flowers, and fruit. Its pharmacological effects that have so far been reported in medical circles include anti-rheumatic, anti-tumor, immunosuppressive, and contraceptive, etc. There have been no reports of it being individually used as a pesticide in agriculture.

The purpose of this invention is to offer a green pesticide against plant-destroying pests using a Tripterygium wilfordii Hook f. extract.

The goal of this invention is achieved in the following manner. The main component of said green pesticide includes extracts TW<sub>I</sub>, TW<sub>II</sub> and TW<sub>III</sub> of the raw medicinal herb Tripterygium wilfordii Hook f. at a weight ratio of TW<sub>I</sub>: TW<sub>II</sub>: TW<sub>III</sub> equal to 1:0.8~1.2:0.8~1.2. The method of preparation of this pesticide calls for combining the extracts of the raw medicinal herb Tripterygium wilfordii Hook f. TW<sub>1</sub>, TW<sub>II</sub>, and TW<sub>III</sub> at a weight ratio of 1:0.8~1.2:0.8~1.2 and then adding other components of Tripterygium wilfordii Hook f. herb to make a mixture. The three components TW<sub>1</sub>, TW<sub>II</sub>, and TW<sub>III</sub> account for 1/500 of the mixture. Alternatively the main component of said green pesticide includes the extracts of the raw medicinal herb Tripterygium wilfordii Hook f. TW<sub>II</sub>, TW<sub>II</sub>, or TW<sub>III</sub>. For the raw medicinal herb Tripterygium wilfordii Hook f. TW<sub>1</sub> extract, the raw medicinal herb Tripterygium wilfordii Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with chloroform to obtain a chloroform extract and subjected to silica gel column chromatography, then eluted with mineral ether: ethyl acetate to obtain the extract TW<sub>I</sub>. It contains five-member unsaturated lactone ring, three-member epoxides, triple six-member rings, hydroxyl groups, and other groups; it is diluted by a factor of 800~1000 with water and its toxic rate with regard to the Wulingcai green worm and Helicoverpa Armigera is as high as 93.6%. For the raw medicinal herb Tripterygium wilfordii Hook f. TW<sub>II</sub> and TW<sub>III</sub> extracts, the raw medicinal herb Tripterygium wilfordii Hook f. is taken, it is percolated and extracted with added ethanol, obtaining ethanol extract, extracted with ethyl ether to obtain an ethyl ether extract, subjected to silica gel column chromatography, then eluted with ethyl ether, to collect fractions containing TW<sub>II</sub> and TW<sub>III</sub>, which then are separately combined, again subjected to silica gel column chromatography, and eluted with chloroform : acetone to collect components TW<sub>II</sub> and TW<sub>III</sub>. They mainly contain a six-member pyridine ring, sesquiterpene lactone, hydroxyl group, and other groups; TW<sub>II</sub> and TW<sub>III</sub> extracts are diluted with water by a factor of 800~1000. Of these components TW<sub>II</sub> has an anti-feeding effect both on Wulingcai green worm and Helicoverpa Armigera, with an anti-feeding rate of 86.7%; TW<sub>III</sub> has a narcotic effect both on Wulingcai green worm and Helicoverpa Armigera with a narcotic

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rate of 89.5%. Due to the agrochemical toxicological combination, as shown by application and follow-up experiments, the three compounds have a unique mechanism of pesticidal effect. The larva, apart from slow movements and manifested lethargy, develop black spots on the surface of their bodies and ever more deformed bodies as well as a number of pathological changes; field experiments on their defensive effectiveness demonstrate a very strong protective effect. Experiments prove that they have a strong pesticidal activity against common pests attacking tea leaves, tobacco leaves, vegetables, cotton, and other cash crops and edible crops. Furthermore, the dose of the three compounds as pesticides is extremely low, and they are not harmful to humans (toxic dose for humans is >10 micrograms/kg, and the dose absorbed after the release of the pesticide is <1 microgram/kg.)

Since this invention uses natural plant Tripterygium wilfordii Hook f. as a pesticide, it cannot release new chemicals into nature or pollute the environment by pesticide residues, thus it falls under chemicals that pose no harm to public health; field experiments on its defensive efficiency also prove that it has a very strong protective effectiveness; its dose as a pesticide is extremely low, and it is not harmful to humans.

Below, we will further explain this invention in combination with Practical Examples:

In the Practical Examples below, the inner diameter of the silica gel column is 6 cm; it was manufactured at the Great Wall Instrument Plant at Zhengzhou. The reagents used are reagents for industrial use. The process of manufacturing the raw medicinal herb Tripterygium wilfordii Hook f. was: Tripterygium wilfordii Hook f. root skin was crushed.

## Practical Example 1

10 kg of the raw medicinal herb Tripterygium wilfordii Hook f. was taken, and, upon adding an appropriate amount of industrial ethanol, it was percolated and extracted, thus obtaining an ethanol extract; this was extracted with an appropriate amount of chloroform, thus obtaining a chloroform extract, and, upon subjecting to silica gel chromatography, it was eluted with mineral ether: ethyl acetate (mol ratio 1:1), yielding the extract TW<sub>1</sub>. It contained It contains five-member unsaturated lactone ring, three-member epoxides, triple six-member rings, hydroxyl groups, and other groups, and as a pesticide, it was diluted with water by a factor of 800~1000; its toxic rate with regard to the Wulingcai green worm and Helicoverpa Armigera was as high as 93.6%.

#### Practical Example 2

10 kg of the raw medicinal herb Tripterygium wilfordii Hook f. was taken, and, upon adding an appropriate amount of industrial ethanol, it was percolated and extracted, thus obtaining an ethanol extract; this was extracted with an appropriate amount of ethyl ether, thus obtaining an ethyl ether extract, upon subjecting it to silica gel chromatography, it was eluted with an appropriate amount of ethyl ether, and fractions of TW<sub>II</sub> and TWIII were collected, the separately combined and again subjected to silica gel chromatography, upon elution with an appropriate amount of chloroform: acetone (mol ratio 1:1), components TW<sub>II</sub> and TW<sub>III</sub> were collected. They mainly contained a sixmember pyridine ring, sesquiterpene lactone, hydroxyl group, and other groups; when

used as pesticides, TW<sub>II</sub> and TW<sub>III</sub> extracts were diluted with water by a factor of \$00~1000. Of these components, TW<sub>II</sub> had an anti-feeding effect both on Wulingcai green worm and Helicoverpa Armigera, with an anti-feeding rate of 86.7%; TW<sub>III</sub> had a narcotic effect both on Wulingcai green worm and Helicoverpa Armigera with a narcotic rate of 89.5%.

# Practical Example 3

The Tripterygium wilfordii Hook f. extract contained extracts  $TW_I$ ,  $TW_{II}$ , and  $TW_{III}$  at a ratio of 1:1:1; the content of the three components  $TW_I$ ,  $TW_{II}$ , and  $TW_{III}$  in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

## Practical Example 4

The Tripterygium wilfordii Hook f. extract contained extracts TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> at a ratio of 1:0.8:0.9; the content of the three components TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

## Practical Example 5

The Tripterygium wilfordii Hook f. extract contained extracts TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> at a ratio of 1.1:1.1:1.2; the content of the three components TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

#### Practical Example 6

The Tripterygium wilfordii Hook f. extract contained extracts TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> at a ratio of 1:0.9:1.2; the content of the three components TW<sub>I</sub>, TW<sub>II</sub>, and TW<sub>III</sub> in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

### Practical Example 7

The Tripterygium wilfordii Hook f. extract contained extracts  $TW_I$ ,  $TW_{II}$ , and  $TW_{III}$  at a ratio of 1:1.1:0.9; the content of the three components  $TW_I$ ,  $TW_{II}$ , and  $TW_{III}$  in the extract was 1/500, and these three components were used as a quality control index. When used as a pesticide, they had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to them.

#### Practical Example 8

The Tripterygium wilfordii Hook f. extract contained extract TW<sub>I</sub>; the content of the component TW<sub>I</sub> in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.

# . Practical Example 9

The Tripterygium wilfordii Hook f. extract contained extract  $TW_{II}$ ; the content of the component  $TW_{II}$  in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.

# Practical Example 10

The Tripterygium wilfordii Hook f. extract contained extract  $TW_{III}$ ; the content of the component  $TW_{III}$  in the extract was 1/500, and this component was used as a quality control index. When used as a pesticide, it had to be diluted with water and appropriate amounts of the solubilizer DMSO and acetone were added to it.